August 2007

Distillation Analysis

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Project Number: **DDB-2371**

DISTILLATION ANALYSIS

A Major Qualifying Project Report

Submitted to the Faculty

of the

WORCESTER POLYTECHNIC INSTITUTE

in partial fulfillment of the requirements for the

Degree of Bachelor of Science

in Chemical Engineering

By:

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Date: **August 10, 2007**

Approved:

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1. batch distillation
2. McCabe-Thiele
3. heat loss

ABSTRACT

The project goal was to optimize the process settings for the multi-stage batch distillation experiment used for CHE2014 to run two experiments per day in accordance with standard three-hour lab periods. The constant molal overflow assumption was questioned and invalidated as means to relate the distillate and bottoms concentrations for the experiment. An alternate theory of incorporating the heat loss effects on the internal reflux ratio at each stage to generate a more accurate operating line was proposed.

ACKNOWLEDGEMENTS

I would like to thank the following people for their help and support to complete this project:

- Amy O'Shea, my wife – thanks for your love and support
- Prof. David DiBiasio, MQP Advisor – thanks for your understanding, patience, and flexibility
- Prof. Anthony Dixon – thanks for your time and input during the mid-term review
- Jack Ferraro and Doug White – thanks for showing me where to find everything, offering your time & experience, and listening to my theories.
- Ryan Kennedy – thanks for sharing your research and reviewing how to run the distillation column
- Andy Peter – thanks for listening to my strategies, sharing your experience from your sophomore year, and working through the data with me. Good luck in your pursuit of your BS in Chemical Engineering
- Christine Love, office of the registrar – thank you for your patience through countless phone calls, emails, and personal meetings to help me re-enter WPI and complete my undergraduate requirements.

TABLE OF CONTENTS

1. EXECUTIVE SUMMARY

The goal of this Major Qualifying Project (MQP) is to improve the quality of the multistage batch distillation experiment used for CHE2014, challenge the theory that the heat loss during the experiment explains the inability to accurately relate distillate and bottoms concentrations using the McCabe-Thiele analysis, and optimize the operating time to allow two complete experiments per day. With the increase in enrollment in the chemical engineering program next academic year, 2007-08, the chemical engineering department would like to find a set of conditions in which the batch distillation experiments can be completed efficiently and effectively.

After the analysis of the prior year's lab reports, a set of conditions was developed to obtain more meaningful results and analysis in the multi-stage batch distillation experiment. Two parameters were discovered that previously had adverse effects to the outcomes. First, an incorrect determination of the minimum external reflux ratio caused students to use external reflux ratios outside the limits of the operating conditions. The resulting operating lines generated were erroneous, and therefore the students were unable to relate the distillate concentration to bottoms concentration via the McCabe-Thiele analysis. Second, due to the azeotropic nature of binary mixtures of ethanol and water, the initial charge contributed to long experimentation times at the azeotrope, and students often obtained few to no samples when the distillate composition was free from the azeotrope. Also found in the student's lab reports, a common explanation for the inability to relate the distillate and bottoms compositions was the poor assumption of constant molal overflow, CMO, due to the heat loss.

Not only did the initial charge restrict the number of experiments per day, the cool down of the column was also a limiting factor. Under the conditions run in D term 2007, the column was required to sit overnight to cool down enough to recharge the column without boiling the ethanol and overflowing the column.

Experiments were conducted to determine the optimal charge concentration to minimize time at the azeotrope, select a balanced external reflux ratio to obtain quality distillate samples off the azeotrope within the limits of the operation conditiond, and develop a cool down procedure, which would allow two complete experiments per day.

An initial charge of 1 wt % of ethanol was found to have an operating time of 45 minutes at the azeotrope. A sample at the azeotrope and 4 samples off the azeotrope were taken easily but require an active team to collect all the information accurately. Based on the vapor-liquid equilibrium data, the minimum external reflux ratio was determined to be 5. An external reflux ratio of 1.5 times the minimum external reflux ratio was used which resulted in optimal operating times while allowing for quality sampling during the experiment. Under the new optimized conditions, experiments were run with and without insulation around the column. Assuming CMO, the linear operating lines used to relate the distillate and bottoms concentrations were determined to be ineffective possibly due to heat loss. It is hypothesized that if the effect of the heat loss at each stage is factored into the determination of the operating line, a curved operating line may allow the distillate and bottoms concentrations to be related accurately.

Two complete experiments per day were realized by finding the optimal processing conditions to relegate the operating time to one standard lab period (3 hours) and developing a shutdown/recharging procedure that could be completed between the

morning and afternoon labs. The shutdown/recharging procedure involved draining the bottoms at the end of the experiment, allowing air-cooling and finally charging the still pot with room temperature water followed by the ethanol.

2. BACKGROUND

The chemical engineering department requires performing a multistage batch distillation experiment for the completion of CHE2014. The assignment given in the most recent class, taught by Prof. Anthony Dixon, can be found in Appendix A.1. The goals of the experiment are the following:

- Conduct a hands-on experimental study of multi-stage batch distillation
- Apply the use of McCabe-Theile analysis, Rayleigh equation, non-steady state material balance considerations, energy balances, validity of CMO, and column efficiency.
- Analyze clearly the performance of the distillation
- Evaluate validity of assumptions made to predict the performance of the distillation
- Make informed and rational recommendations for the future use of the column

In the following section, an explanation of the pre-lab preparation and post-lab analysis will be detailed. The same structure will be later used in the Results and Discussion of this project.

2.1 Pre-Lab Preparation

Prior to the lab, students are required to identify the experimental data to collect, prepare the settings for experiment, create a sampling procedure, and make predictions of the outcomes based the given information provide in the assignment.

Identifying the data to collect during the experiment is critical to analyzing the performance of the separation and column. For the McCabe-Thiele analysis, samples, and their temperatures at sampling, of the distillate and bottoms products are required. To perform an overall energy balance, the temperatures, in and out, and flow rates of the steam and cooling water are required. Using the energy balance, the students can evaluate the validity of the assumption of constant molal overflow, CMO. "CMO is when molar vapor, V, and liquid, L, flow rates in each section are constant." Wankat [1] The condition of constant molal overflow requires the following assumptions to be true, Wankat [2]:

1. Well-insulated column
2. Constant heat capacities and sensible heat changes small compared to latent heat changes
3. Same latent heat of vaporization per mole for the two components

Since L and V are constant, the operating equation that relates the concentrations of two passing streams in column becomes Wankat [3]

$$y = \frac{L}{V}x + \left(1 - \frac{L}{V}\right)x_D$$

The settings for the experiment that need to be determined include the initial charge of ethanol in pot, steam pressure, cooling water flow rate, external reflux ratio, R. With the exception of the external reflux ratio, an acceptable range of values for the settings should be given to the students in order maintain experimental quality and consistent operating time. The external reflux ratio is determined from the limits of the operating conditions.

"The limits of the operating conditions are total reflux and minimum reflux" Wankat [4]. For a batch distillation at total reflux, all distillate and bottoms flows are returned to the column. There are two uses for running a distillation column at total reflux. One, when starting up a column, total reflux is an effective setup to obtain a steady state within the column prior to taking product and/or introducing feeds. Second, total reflux is used for evaluating the overall column efficiency.

Using the material balance around the condenser,

$$V = L + D$$
$$Vy = Lx + Dx_d$$

at total reflux all of the vapor that is condensed and returned to the top stage. Therefore, distillate flow rate, D, equals 0, and the vapor flow rate into the condenser, V, is equal to the liquid flow rate back into the top stage, L. Under this condition, the operating line is y = x. "Total reflux gives the minimum number of stages required for a given separation." Wankat [5]

The other limit is the minimum reflux ratio, R_{min}; this condition is "the external reflux ratio at which the desired separation could just be obtained with an infinite numbers of stages". Wankat [6] An infinite number of stages can be obtained when the operating and equilibrium lines are tangential or meet. Binary mixtures of ethanol and water contain an azeotrope. An azeotrope is a blend of two or more components whose equilibrium vapor phase and liquid phase compositions are the same at a given pressure. Using the distillate concentration at the azeotrope, an operating line can be drawn that is tangential to the equilibrium line. The slope of this operating line is the minimum internal reflux ratio, $(L/V)_{min}$, and the R_{min} can be determined from the following equation:

$$R_{\min} = \left(\frac{L}{D}\right)_{\min} = \left(\frac{L}{(V-L)}\right)_{\min} = \frac{\left(L/V\right)_{\min}}{1-\left(L/V\right)_{\min}}$$

So, the external reflux ratio to be used in the experiment must lie between R_{min} and total reflux. It was found in the lab reports from D term that students had been given a value for R, which create an operating line that immediately crossed the equilibrium line. The resulting operating lines do not allow the distillate composition, x_D, and bottoms composition, x_w, to be related. This is demonstrated in Fig. 2.1.

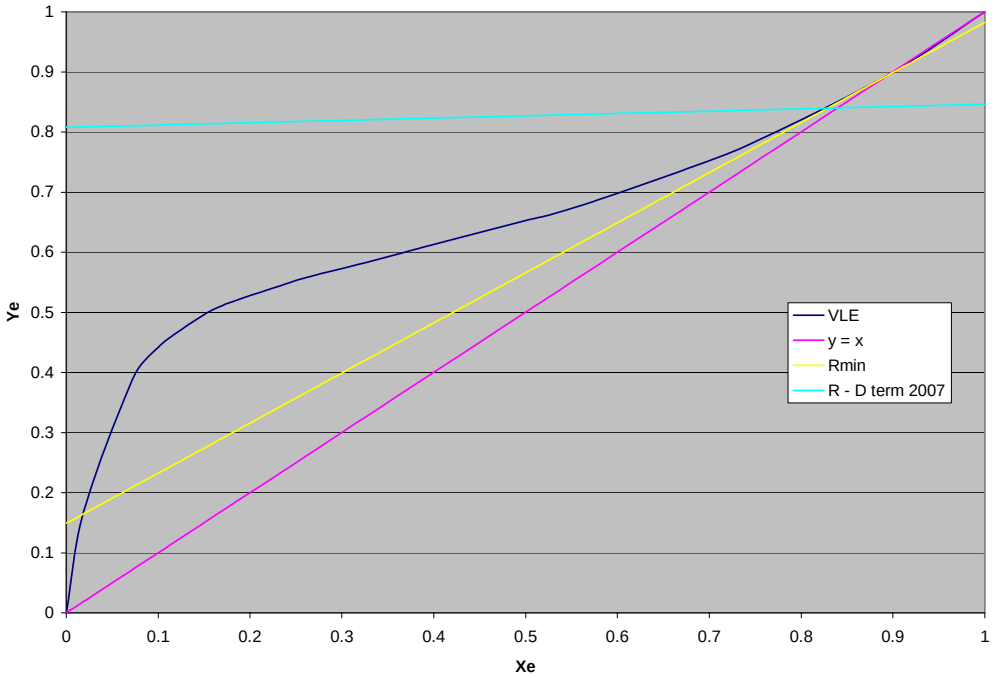


Figure 2.1 McCabe-Thiele diagram ethanol-water at 1 atm - Comparison of operating lines used on D term 2007 versus operating lines generated from Rmin and total reflux

The external reflux ratio can be specified by using a multiple of R_{min}. For example, 1.5 R_{min} was used for the experiments in this project.

Next, the students must create a sampling procedure. This procedure should include the number of samples, process for taking the samples including safety equipment, and reasoning behind the sampling process. It is important for students to clearly understand that temperature and composition are directly related. Obtaining samples off the azeotrope, the students will be able to relate the distillate concentration to the bottoms concentration at various stages in the separation until they reach the predicted value of the final distillate composition, $x_{D,final}$, which signals the completion of the run.

Finally, students are asked to make predictions of the outcome of the separation. Mass balances around the entire column are

$$F = W_{final} + D_{total}$$

$$Fx_f = W_{final}x_w + D_{total}x_{D,avg}$$

From the lab assignment found in Appendix A1, given the goal of collecting 80% of the initial total ethanol, "predictions of the amount (moles), D_{total}, and composition (mole fraction) of the distillate, $x_{D,final}$, obtained when the goal is reached, the average distillate composition for the run, $x_{D,avg}$, and the amount (moles), W_{final}, and final composition, $x_{w,final}$, (mole fraction) of the still pot" can be calculated. Since the assumption of CMO provides a method for relating the distillate concentration, x_D, to the

bottoms concentration, x_W, the Rayleigh equation can be used to determine W_{final}, $x_{w,final}$, and $x_{D,final}$.

$$\ln\left(\frac{W_{final}}{F}\right) = -\int_{x_{w,initial}}^{x_{w,final}} \frac{dw}{x_D - x_W}$$

$$1 - \frac{W_{final} x_{w,final}}{F x_f} = \%_{recovery}$$

Using the two above equations, it is necessary to use an iterative method to find W_{final}, $x_{w,final}$, and $x_{D,final}$. The average distillate composition for the run, $x_{D,avg}$, is then determined from the material balance.

$$x_{D,avg} = \frac{F x_f - W_{final} x_{w,final}}{F - W_{final}}$$

Optimal pre-lab preparation will include a spreadsheet containing all the variables to record along with detailed procedure of what each team member will do during the experiment. The settings should be reviewed and approved ahead time to make sure that not only do the students clearly understand the concepts, but that the experiment produces quality results within the given lab period.

2.2 Post-Lab Analysis

As stated in the lab assignment found in Appendix A1, "the goal is to analyze the column's performance given an initial charge to the still pot and a goal of recovering 80% of the initial total ethanol, and make informed and rational recommendations for the column's future use." The post lab analysis should include the following:

1. Evaluation of the overall column efficiency
2. Calculation of the heat loss and affects to CMO assumption
3. Evaluation of predicted values versus measured values of distillate and bottoms accumulations and concentrations along with along with operating time to achieve the goal
4. Recommendations on how to improve the overall use of the column

The overall column efficiency, E_o, is calculated by

$$E_o = \frac{N_{equil}}{N_{actual}}$$

where N_{equil} is the number of equilibrium stage required for the separation and N_{actual} is the number of stages in the column. Wankat [7] Once the reflux meter is started, samples of the distillate and bottoms should be taken. Using the McCabe-Thiele diagram,

equilibrium stages can be stepped off from x_D to x_W between the operating line of y = x and the VLE line.

Each assumption of CMO should be questioned to confirm that CMO is useful for relating x_D and x_W and hence the predictability of the column's performance. The CMO assumption allows a linear operating line to be generated providing an easy method for relating x_D to x_W. Using the Rayleigh equation, a prediction of final column conditions at the given goal can be determined. Currently, the column is not well-insulated so the students may see the activity at each stage. Calculating the heat loss on the column will put the validity of the CMO assumption into perspective. The energy balance for the system, assuming all kinetic and potential energy changes are negligible, is

$$Q_C + Q_R + Q_{loss} + Dh_D = 0$$

Q_C, the heat flow of the condenser, is determined by:

$$Q_C = \dot{m} C_P \Delta T$$

The heat capacity of water, C_P, is assumed to be constant over the temperature range. Q_R, the heat flow of the reboiler, is determined by:

$$Q_R = \dot{m} \Delta H$$

The enthalpy change of steam can be determined from the steam tables. The condensate is assumed to be saturated liquid at atmospheric pressure. Finally, the enthalpy of the distillate, h_D, can be determined from the enthalpy-composition diagram for ethanol and water (Wankat [8]) and can be assumed to be constant throughout the experiment.

Evaluation of the predicted values versus actual values is the starting point for the recommendations of column's future use. If the current assumption of CMO does not allow adequate predictability of the outcome of the separation, a new set of assumptions should be determined. In order to satisfy the goals of the experiment, the students must understand how they can improve the predictions and actual performance of the separation based on their assumptions and what affected those assumptions.

The recommendation for future use of column should be focused on the ability of the column to perform the separation and how the performance can be improved. Key variables to consider should include optimizing total operating time, energy consumption, and predictability of unit operation performance.

3. METHODOLOGY

3.1 Lab Safety

- Wear goggles and hard hats at all times during the lab.
- Review Material and Safety Data Sheet, MSDS, on ethanol.
- Use asbestos gloves when taking samples of bottoms, steam condensate and/or disposing of bottoms.

- During off-hour operation, two people are required in the lab at all times

Table 3.1 Process Conditions for Experiments

Process Variable	Experiment 1	Experiment 2	Experiment 3	Experiment 4	Experiment 5
$x_{wintial}$	6%	3 wt%	1 wt%	1 wt%	1 wt%
P_{steam}	8 psig	10 psig	10 psig	10 psig	10 psig
R	0.2	7.5	7.5	7.5	Total Reflux; R = 7.5
Cooling Water Flowrate	90%	90%	90%	90%	90%
Insulation on Column	Yes	Yes	Yes	No	Yes

3.2 Startup Procedure

1. Turn on cooling water and set flow meter to desired value according to Table 3.1.
2. Charge with a mixture of ethanol and water according to Table 3.1.
3. Confirm reflux meter is off.
4. Purge steam line of condensate by opening condensate value until only steam is released.
5. Open steam flow value full.
6. Close steam pressure value until the desired steam pressure is obtained according to Table 3.1. (Maximum P_{steam} = 14 psig)
7. Wait until temperature at the top stage finds equilibrium.

3.3 Sampling Procedure

1. Set reflux meter to desired value according to Table 3.1
2. Turn on reflux meter.
3. Take samples
    a. Record time
    b. Record steam pressure, P_{steam}.
    c. Record temperature of steam in, $T_{steamin}$.
    d. Measure steam flow rate, g/min, by collecting condensate out
    e. Record cooling water flow rate.
    f. Record temperature of cooling water in, T_{cwin}.
    g. Record temperature of cooling water out, T_{cwout}.
    h. Record temperature of bottoms, T_B.
    i. Take sample of bottoms, and allow sample to cool to room temperature.
    j. Measure density of bottoms sample, and record, x_B
    k. Record temperature at top stage, T_{top}.
    l. Open distillate value and allow collected distillate to accumulate in large graduated cylinder
    m. Measure distillate flow rate, g/min, while collecting distillate
    n. Measure density of distillate sample, and record, x_D
4. Repeat step 4 for additional samples allowing T_{top} to change at least a full degree Celsius before taking another sample. Stop taking samples once $x_{D,final}$ is reached.
5. At the end of the experiment, after distillate and bottoms samples have been measured, the samples should be combined with the total distillate or bottoms.

6. Measure and record the mass, kg, and density of the total distillate
7. Measure and record the mass, kg, and density of the total bottoms

3.4 Shutdown/Recharging Procedure

1. Turn off reflux meter
2. Turn off steam to reboiler by opening steam pressure value
3. Close steam flow value
4. Measure and record total remaining mass of bottoms.
5. Drain bottoms in sewer drain. Caution bottoms are at ~99ºC.
6. Allow column to air cool for 1 hour. The cooling water should be running throughout.
7. Check temperature of bottoms. If $T_B < 60$ ºC, charge distillation pot with required water.
8. Check temperature of bottoms. If $T_B < 30$ ºC, charge distillation pot with required ethanol.
9. If no other experiments are scheduled, turn off cooling water.

4. APPARATUS

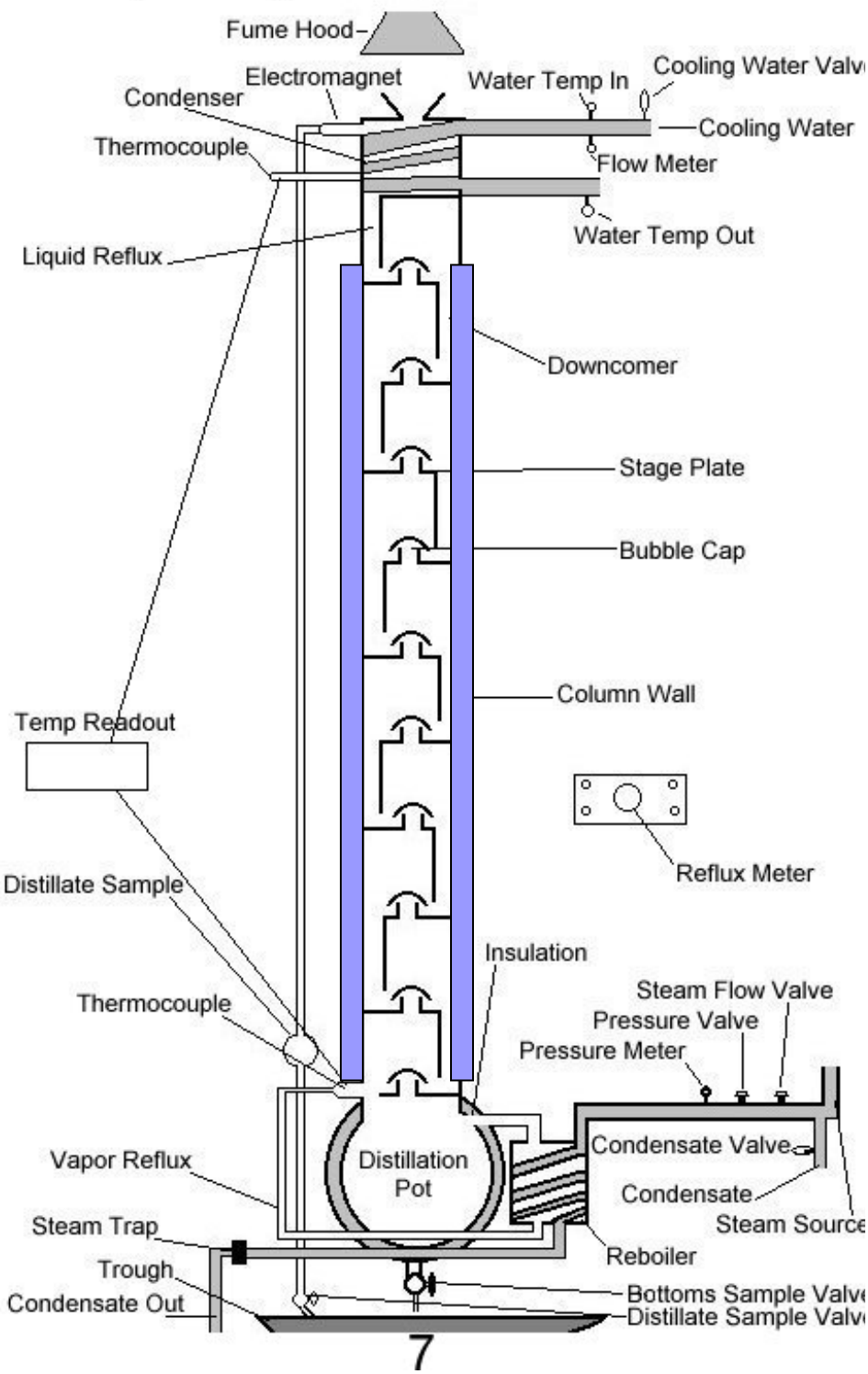


Fig. 4.1 Ten Stage Bubble-Cap Distillation Column

5. RESULTS & DISCUSSION

The set of experiments run were selected to improve the quality and efficiency of the batch distillation experiment. The goals of the first three experiments were to find the proper balance of process variables to obtain a complete distillation within a standard 3-hour lab period and improve the quality of the experiment so that the measured values can be easily compared to predicted values. The fourth experiment was a replicate of experiment three and will be used to demonstrate the effectiveness of the improvements by following the same analysis used in the lab assignment for CHE2014. Experiment 5 was an experiment to verify the stability of holding a certain distillate concentration just outside the azeotrope for the total reflux experiment for CHE2012.

5.1 Experiments 1-3

Initially, the primary goal of experiment 1 was to review how to run the column safely and effectively. The experiment was conducted under the instruction of Ryan Kennedy, who had recently completed an MQP involving the distillation column. As seen in Table 5.1, the process settings were chosen based on prior research conducted by Ryan Kennedy. Also, in review of the lab reports from D term 2007, the students of CHE2014 ran similar settings.

Table 5.1 Experiment 1 - Process Settings and Results

Process Variable	Experiment 1
x_f	~6%
P_{steam}	8 psig
R	0.2
$t_{startup}$	53 min
$t_{azeotrope}$	1 hrs 45 min
# of samples after azeotrope	0
t_{oper}	6 hrs
% recovery	N/A

Two important issues were revealed during this experiment. First, R is outside the limitations for a binary mixture of ethanol and water. As demonstrated by Figure 2.1, $R_{min} = 5$ and therefore all possible R's for the experiment must be greater than R_{min}. Choosing an $R \leq 5$ adds significant error to the predictions of the final outcomes of the experiment as determined by the Rayleigh equation, and in most cases it was not possible to calculate the predictions because it is not possible to accurately relate x_D to x_W.

Second, the operating time, t_{oper}, was twice the desired experimental time. During the experiment, the students are required to run the experiment until the $x_{D,final}$ is

achieved. So, in order to reduce the overall experiment time, it is necessary to reduce the amount of time spent at the azeotrope. Since the distillate concentration will not leave the azeotrope until the total amount of ethanol is low enough in the column, the initial charge of ethanol, x_f, must be reduced to shorten the time at the azeotrope. As result of identifying these two issues, the process settings for experiment 2 is shown in Table 5.2

Table 5.2 Experiment 1 and 2 - Process Settings and Results

Process Variable	Experiment 1	Experiment 2
x_f	~6%	3%
P_{steam}	8 psig	10 psig
R	0.2	7.5
$t_{startup}$	53 min	1 hr
$t_{azeotrope}$	1 hrs 45 min	3 hrs 49 min
# of samples after azeotrope	0	3
t_{oper}	6 hrs	5 hrs 45 min
% recovery	N/A	N/A

The steam pressure was increased to 10 psig to shorten startup time, but no change was observed. The affect of changing R from 0.2 to 7.5 ($1.5R_{min}$) was a slower distillate flow rate. Prior to experiment 2, it was hypothesized that the reduction of x_f from 6% to 3% would offset the change in the external reflux ratio, and therefore the time at azeotrope would be similar. However, in experiment 2, the time spent at the azeotrope was much longer that experiment 1; this suggested the initial charge of ethanol would again need to be reduced for experiment 3.

In experiment 3, the optimal process settings were achieved to obtain a complete experiment in a three hour lab period, as shown in Table 5.3. By reducing the initial charge to 1% ethanol, the resulting total operating time was approximately 3 hours. Five samples off the azeotrope were successfully taken.

It was hoped to use the data from experiment 3 to compare predicted versus measured values, but during the experiment, the condensate flow rate was not measured or recorded and the total distillate was spilled during measuring. So, experiment 4 was conducted as a replicate of experiment 3 to obtain high quality samples to complete a quality comparison of the predicted versus measured values at the optimal process settings.

Table 5.3 Experiment 2 Process Settings and Results

Process Variable	Experiment 1	Experiment 2	Experiment 3
x_f	~6%	3%	1%
P_{steam}	8 psig	10 psig	10 psig
R	0.2	7.5	7.5
$t_{startup}$	53 min	1 hr	1:25 min
$t_{azeotrope}$	1 hrs 45 min	3 hrs 49 min	45 min
# of samples after azeotrope	0	3	5
t_{oper}	6 hrs	5 hrs 45 min	2 hrs 56 min
% recovery	N/A	N/A	N/A - spilled

5.2 Cool Down Procedure

Between experiments, a procedure was developed to minimize the setup time of the column. Prior to this project, after an experiment was completed, the reflux meter and steam were turned off, and the column sat over night for the bottoms to cool down; this procedure only permitted one experiment per day. From discussions with Jack Ferraro, a faster procedure has never been evaluated in the past.

At the end of a batch distillation experiment, the still pot contains a mass of about 45- 50 kg at a temperature of approximately 99 ºC. If ethanol were added to the column at his point, it would boil and cause the column to overflow. The fastest way to cool down the column was to eliminate this heat sink and recharge the column with fresh materials. The concentration of the ethanol is so small at ~99 ºC that the bottoms can be safely drained into sewer without any risk to the students or sewage system. The students or school personnel should wear asbestos gloves when draining the bottoms to the sewer.

With the heat sink removed, the column cooled down quickly. After 1 hr with only the cooling water running, the temperature in the still pot in the empty column went from ~99 ºC to ~60 ºC. At this point, room temperature distilled water was added to the still pot; by the end of the charging of water, the temperature in the still pot had dropped below 30 ºC. Following this procedure, the ethanol could be safely added. The cool down time including recharging was ~90 minutes, which is enough time to prepare a fresh charge between morning and afternoon labs (~120 minutes).

5.3 Experiment 4: Optimal Process Settings

Experiment 4 was run under same process conditions as experiment 3 with one exception; the insulation around the 10 stages of the column was removed. The purpose of removing the insulation was to allow the students to see the separation at each stage during the experiment.

5.3.1 R Determination

$R = 1.5R_{min}$ was initially chosen arbitrarily to generate an operating line that was within the limits of the operating conditions, R_{min} and total reflux. During experiment 2, the chosen R was to determined to be appropriate for the project based on the impact on the length of total operating time (standard 3 hour lab period), and the ability for the students to take accurate distillate samples at a particular distillate temperature, T_D. From experiment 4, the time required to collect the total amount distillate, $t_{operating}$, was calculated by

$$t_{operating} = \frac{D_{total}}{D} = \frac{465.56g}{5.63g/\min} = 82.8\min$$

This $t_{operating}$ corresponds to about one half of a standard lab period. Also during experiment 2, samples of the distillate were taken at no change in T_D, thus ensuring that the measured values of x_D and T_D corresponded.

5.3.2 Temperature Correction

The measured temperature at the top stage of the first sample was outside the VLE data for the binary mixture, probably due to the lack of insulation around top stage and the resulting heat loss to the condenser. A temperature correction factor was developed by taking the x_D and x_W pair immediately measured after the reflux meter was started and finding the associated VLE temperature. The correction factor was added to the temperature of each sample.

$$T_{theoretical} - T_{Experimental} = T_{CorrectionFactor}$$

For experiment 4, the correction factor was 0.76 ºC.

5.3.3 Determination of Distillate and Bottoms Concentrations

The proper procedure for this analysis is to measure the concentrations by density or specific gravity and then convert to concentration. The device provided by the chemical engineering department that measures the density was out for repair, therefore the compositions were determined from the temperature-composition diagram generated from the VLE data.

5.3.4 Column Efficiency

The number of equilibrium stages was determined from stepping off stages on a McCabe-Thiele diagram using x_D and x_W immediately after the reflux meter was started. See Appendix A.2

$$N_{Equilibrium} = 6$$

$$Eo = \frac{N_{Equilibrium}}{N_{Actual}} = \frac{6}{10} = 60\%$$

5.3.5 Determination of Heat Loss and Validity of the CMO Assumption

Table 5.4 Energy Balance Calculations

Q_c	Q_r	Dh_D	Q_{loss}	% Q_{loss}
kJ/min	kJ/min	kJ/min	kJ/min	
-230.99	271.28	-1.30	-41.59	-15%

As show in table 5.4, the heat loss was about 15% of the heat input by the reboiler. The assumption of a well-insulated column is close but not accurate and thus calls into question the CMO assumption and any predicted values from the Rayleigh equation.

Furthermore, demonstrated by Fig 5.1, x_D and x_W can not be related because the operating lines, created from the CMO assumption, cross the VLE line at x_E higher than the initial concentration, $x_f = .0039$..

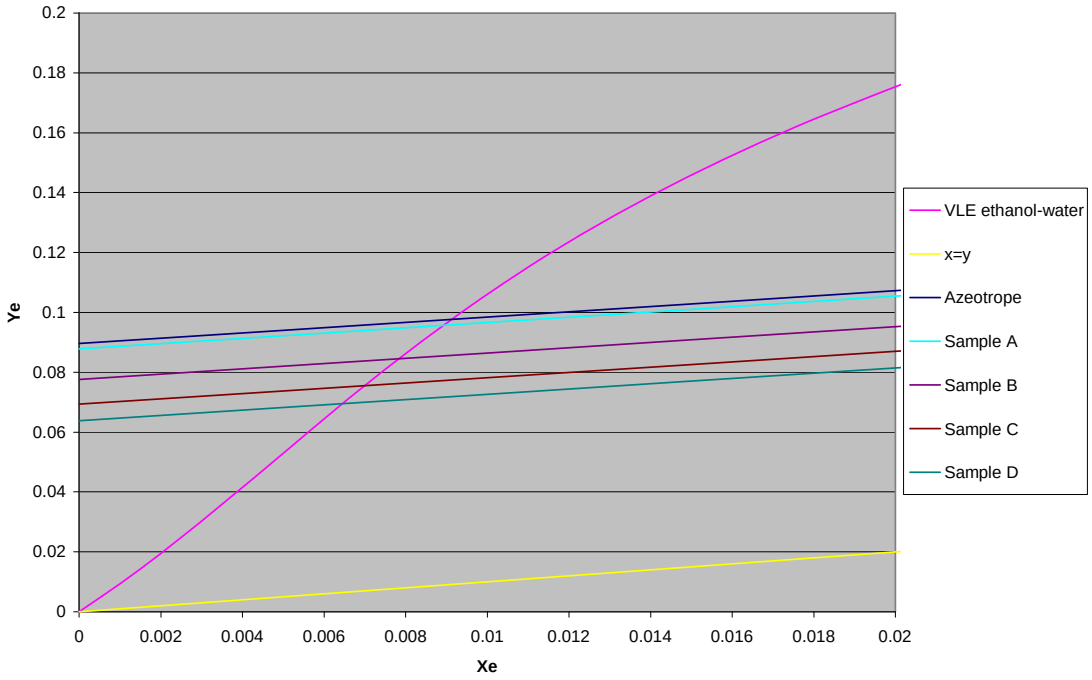


Fig 5.1 McCabe-Thiele diagram ethanol-water at 1 atm - Experiment 4, examination of operating lines generated under the assumption of CMO

An alternative method for relating x_D and x_W may be to generate an operating line that takes into consideration the heat losses on the column. If the column were well insulated, all the heat must be removed by the condenser. For the column during experiment 4, the still pot was insulated but the all the stages above the reboiler were not insulated. It is safe to assume that the heat loss is the greatest at the first stage above the reboiler and rate of heat loss decreases as the distance between the stage and the reboiler increases.

According to R.E. Treybal, “heat loss increases the internal-reflux ratio.” Treybal [9] Assuming the top stage most closely represents the behavior at CMO and each stage under the top stage has a linear increase in internal-reflux ratio from one stage to the next approaching the boiler, the resulting operating line would have a progressively steeping slope as presented in figure Fig 5.2.

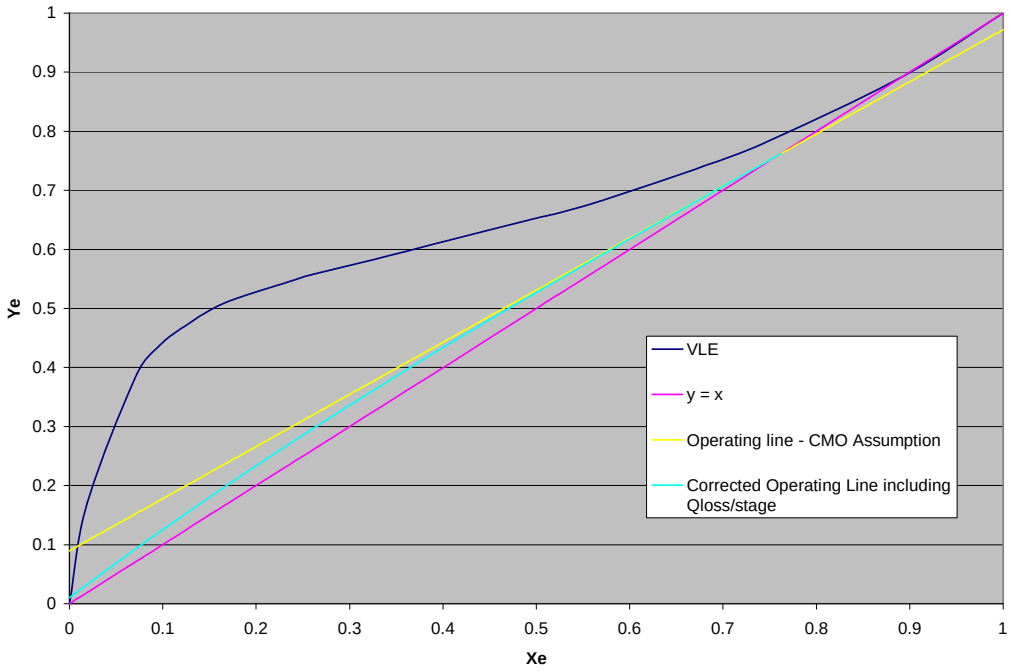


Fig 5.2 McCabe-Thiele diagram ethanol-water at 1 atm - Exp. 4, comparison of the operating line generated via the CMO assumption versus operating line incorporating heat loss

As shown in Fig 5.3, the corrected operating line can allow the relation between x_D and x_W because the stepped off stages can reach the values $x_W < x_f$. The Rayleigh equation could then be employed with the new data to predict the outcomes more accurately.

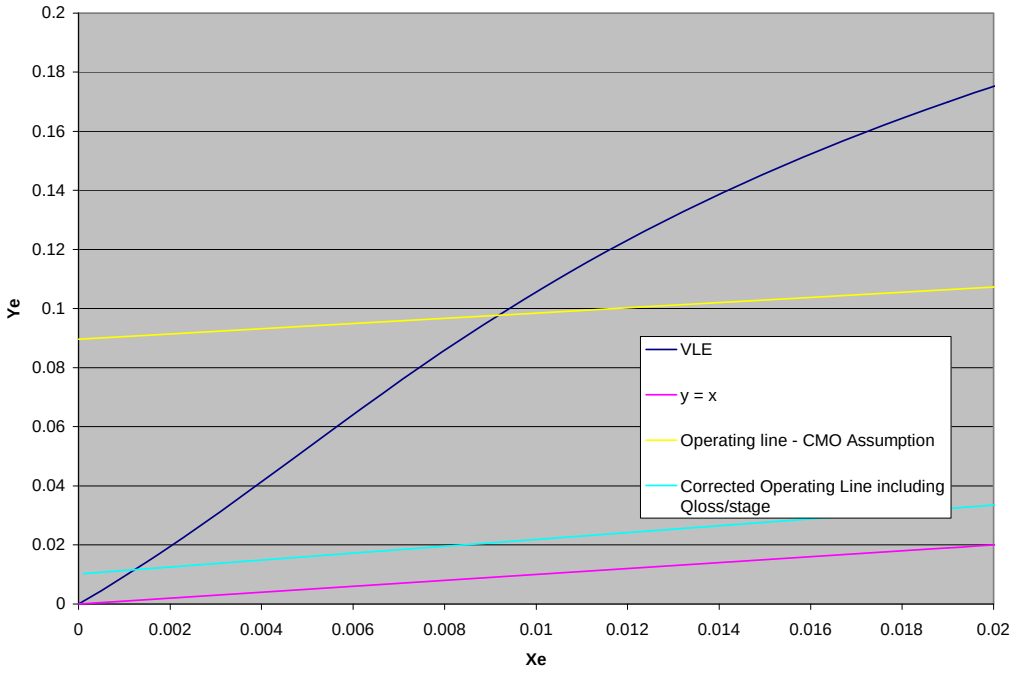


Fig 5.3 McCabe-Thiele diagram ethanol-water at 1 atm - Exp. 4, comparison of the operating line generated via the CMO assumption versus operating line incorporating heat loss

5.3.6 Evaluation of the Rayleigh Equation Prediction versus Measured Values

The theoretical values determined from the Rayleigh equation were completed after the completion of experiment 4. The reason for this was to use the actual % recovery from experiment 4 for the goal of the predicted values. The results are displayed in Table 5.5.

Table 5.5 Comparison of the Predicted outcomes of experiment 4 versus measured

	F	W_{final}	D_{final}	$x_{D,\ final}$	$x_{D,\ avg}$	$x_{W,\ final}$	% recovery
	mol	mol	mol				
Predicted	2.9	2.84	0.06	0.082	0.1317	0.0012	70%
Actual	2.9	2.89	0.01	0.542	0.614	0.0034	71.4%

The primary reason for the lack of accuracy of the predicted values stems from the inability to relate the x_D and x_W. Table 5.6 was created by using the measured samples of x_D, using the operating lines with R = 7.5 assuming CMO, and stepping of the number of equilibrium stages to obtain corresponding x_W.

Table 5.6 Predicted values, based CMO assumption, of x_W using measured values of x_D

x_D	x_W
0.762	0.0091
0.746	0.0090
0.660	0.0076
0.589	0.0072
0.542	0.0067

All of the corresponding values of x_W are above the initial charge concentration, x_f = 0.0039. Therefore the predicted values are erroneous, and the assumption of CMO is invalid.

5.4 Experiment 5

The goal of experiment 5 was to verify the stability of holding x_D at total reflux where x_D is off the azeotrope. The process conditions were the same as experiments 3. Distillate was taken until T_D had increased 1.0 ºC from the initial T_D. The column was then put into total reflux and T_D monitored. During one hour and 13 minutes at total reflux, T_D increased 0.4 ºC. The value of this understanding is teams of students conducting the total reflux lab in CHE2012 can obtain near azeotrope concentrations to determine the number of equilibrium stages and therefore the overall column efficiency.

6. Recommendations

Recommendations for further improvement of the operation of the distillation column include:

- Connect a de-ionized water feed to the still pot for easy charging. Currently, the water must be charged by hand
- Insulate top stage to improve the accuracy of the T_D reading.
- Use sensitive measurement method to detect low concentrations ethanol in samples

Recommendations for further research include:

- Upgrade column with real-time computer-monitoring and control systems
- Quantify the effect of heat loss on the internal reflux ratio stage by stage
- Develop a more accurate model to predict the performance of the separation of binary mixture of ethanol-water.

APPENDIX A1

CHE 2014 PROJECT 2:

MULTISTAGE BATCH DISTILLATION

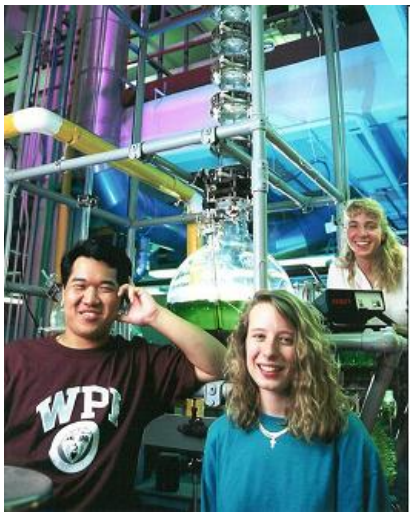
To: *CHE 2014 Design Teams*
From: A.G. Dixon, Vice President
Subject: Assessment of Pilot Plant Column
Date: April 2, 2007

Introduction

Our company needs to evaluate the pilot plant multistage batch distillation column located in our Goddard Hall facility. Your team will analyze the column's performance given an initial charge to the still pot and a goal of recovering 80% of the initial total ethanol. This pilot study includes experimental work, an analysis, and a written report. The schedule is tight - we need each team's report by 5 pm on April 19. The lab runs of the column can be lengthy, and it can be difficult to maintain sampling duties throughout a day. For this reason, we will combine teams for the experimental part of the project only. Each of the original teams is still responsible for writing their own report, but pairs of teams will share data.

Details of Pilot Plant Work

For this project, you have available the glass distillation column in the Unit Operations Lab and other resources you may need. The distillation column is pictured here, along with distinguished former company engineers. Recall that the column is equipped with:



- sample ports for the reboiler and distillate,
- temperature measurements throughout the system,
- sample ports on each stage,
- reflux ratio adjustment mechanism,
- specific gravity instrument for ethanol analysis,
- steam condensate flow,
- steam pressure measurement,
- condenser flow rate and temperatures,
- stopwatch for timing reflux ratio, distillate flow rate, and steam condensate rate.

Your task includes choosing an appropriate reflux ratio; predicting the amount (moles) and composition (mole fraction) of the distillate obtained when the production goal is reached; the averaged distillate composition for the run; and the final composition and total amount remaining in the still pot. Assuming a constant boil-up rate, compare experimentally obtained column transients to predicted ones. Finally qualitatively (or quantitatively, if possible) explain any differences. There are a number of topics that may

be important in your analysis. These include application of the Rayleigh equation, nonsteady-state material balance considerations, energy balances, validity of CMO, and column efficiency. Your conclusions should include an informed and rational recommendation for the column's future use in our context.
Your team(s) will need to think out the experiment in detail before making a run. The lab can be completed in 4-5 hours with an informed choice of steam pressure and external reflux ratio. However, you might easily be there 8-10 hours if you make little or no pre-lab preparation. To avoid problems, you are required to hand in a Pre-lab report by Monday, April 9th. This should be 1-2 pages, no background or introduction, and should indicate your choice of settings for the experiment, which quantities you will sample and how often and which experimental data you will use to check your calculations and assumptions about the column.

We will schedule lab times as the project develops. Please refer to the posted project team list so you can connect with a team and reserve a joint lab time. Remember that hard hats and safety glasses are required when in the lab.

Lab. Guidelines

1. Data will be collected by two teams at once. You may need up to 10 hours to get all the data.
2. During normal business hours (8-5) one person can be present to take samples and data. Before 8am or after 5pm requires two people to be present.
3. Sampling every 30-60 minutes will generally be sufficient, although near the end of the run you may want to sample a little more frequently.
4. Each team should discuss and plan their experiment then elect a representative (or two) to communicate with the partner team about the joint experiment. It is not necessary, nor is it productive, to have an 8-person meeting to finalize lab arrangements.
5. External reflux ratio choice is important for estimating total lab time. You can assume column efficiency is constant during the run. Our electronics engineer, Doug White, is available to show you how to use the reflux control mechanism.

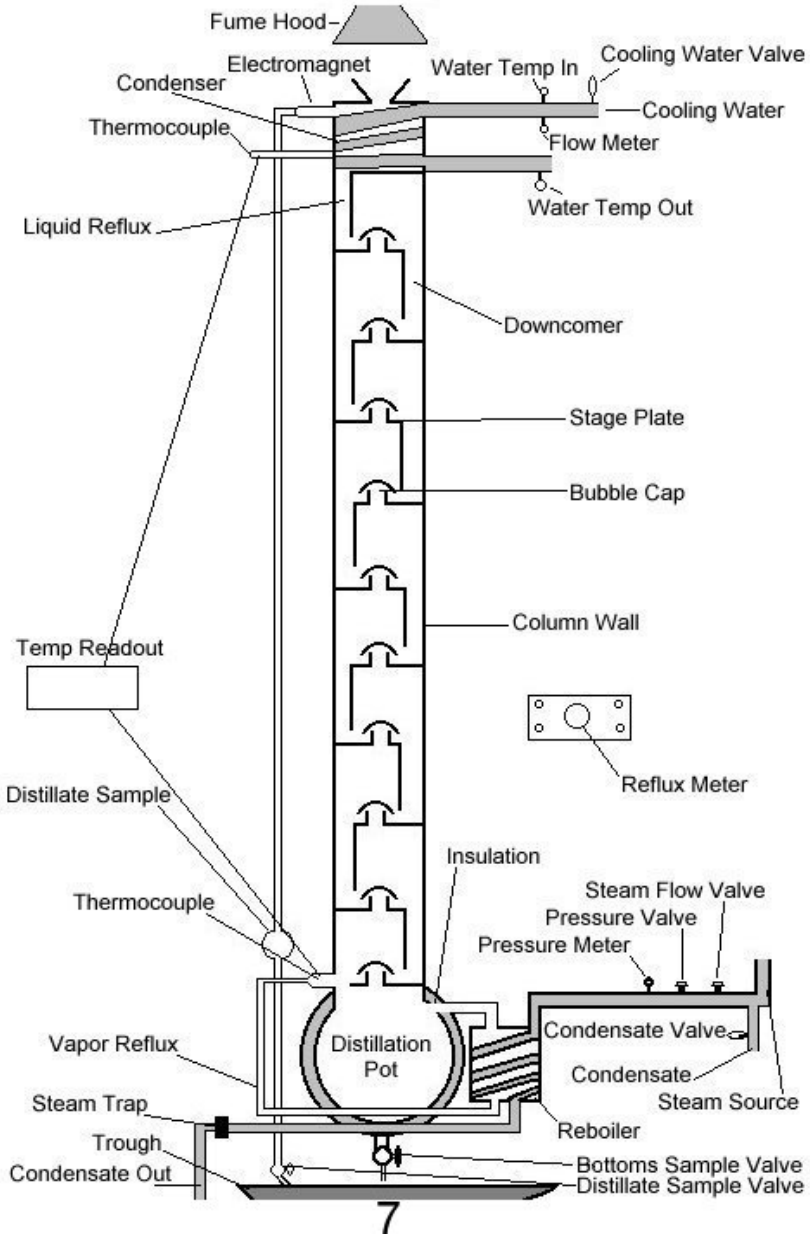
Report Guidelines

Your report should follow the format below:

1. Letter of transmittal: on your team's letterhead
2. Introduction: Include some background and the specific objectives of your study
3. Methods: Describe the pilot study protocol in detail, including a drawing of the equipment and specific methods employed. Include a discussion of safety precautions that you took.
4. Results and Discussion: This is where you present important results and discuss their meaning. Put important plots, tables, results of calculations, figures, etc in this section. Do not bury them in the appendix.
5. Conclusions and Recommendations: Describe these as appropriate for your work and this assignment.

6. Appendix: include references, raw data, detailed calculations and spreadsheets, etc. Important results from calculations should be reproduced in the Results and Discussion session and presented there in a visually appealing way. The appendix is also a suitable place to put any reference data that you obtained from other sources, including safety information.

Fig. 1 - Ten Stage Bubble-Cap Batch Distillation Column



Safety Precautions

- Denatured → Defined as made **unfit for eating or drinking** without destroying its usefulness for other purposes. The ethanol in the tower is DENATURED with benzene.
- Hard hats and eye protection are required at all times.
- Be familiar with the MSDS for ethanol. (http://www.wpi.edu/Admin/Safety)
- Asbestos gloves must be worn during bottoms and steam condensate sampling.
- DO NOT touch steam lines or glass surfaces with bare hands.
- DO NOT drink the product from the tower. Consequences would be dire.
- DO NOT drop your hard hats or anything else on people below when working on second floor of the laboratory.

Safety Contacts

- Prof. Dixon → GH224A, x5350, agdixon@wpi.edu
- Jack Ferraro (GH Shop Manager) → GH09B, x5237, gferraro@wpi.edu
- David Messier (WPI Safety Officer) → OH030, x5216, dmessier@wpi.edu

- ***Know the location of all safety showers, eye wash stations, and fire extinguishers.***

APPENDIX A2 Graph - Column Efficiency

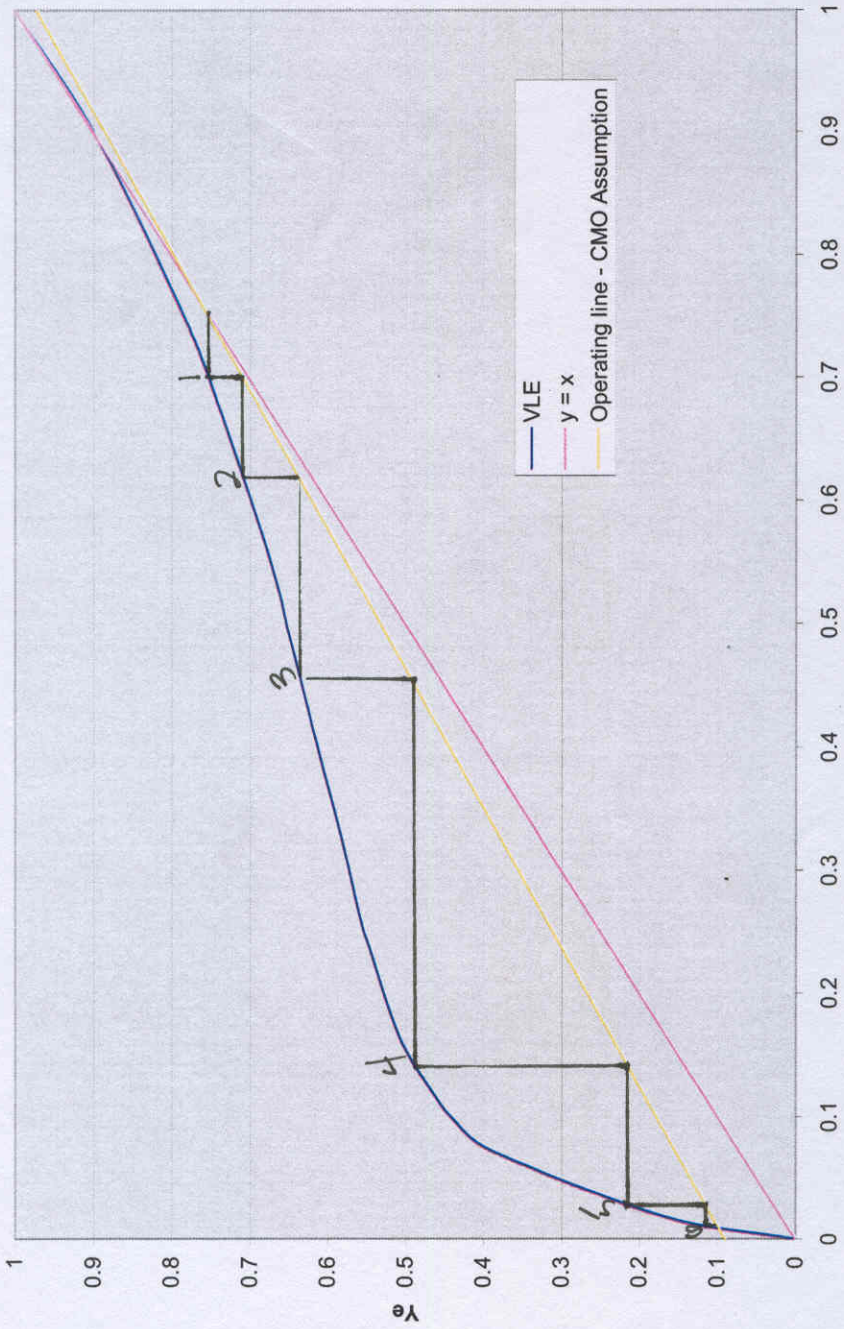


Fig A2.1 McCabe-Thiele diagram ethanol-water at 1 atm - Exp. 4, Determination of the number of equilibrium stages for column efficiency

APPENDIX A3 Graph – Determination of x_D via CMO assumption

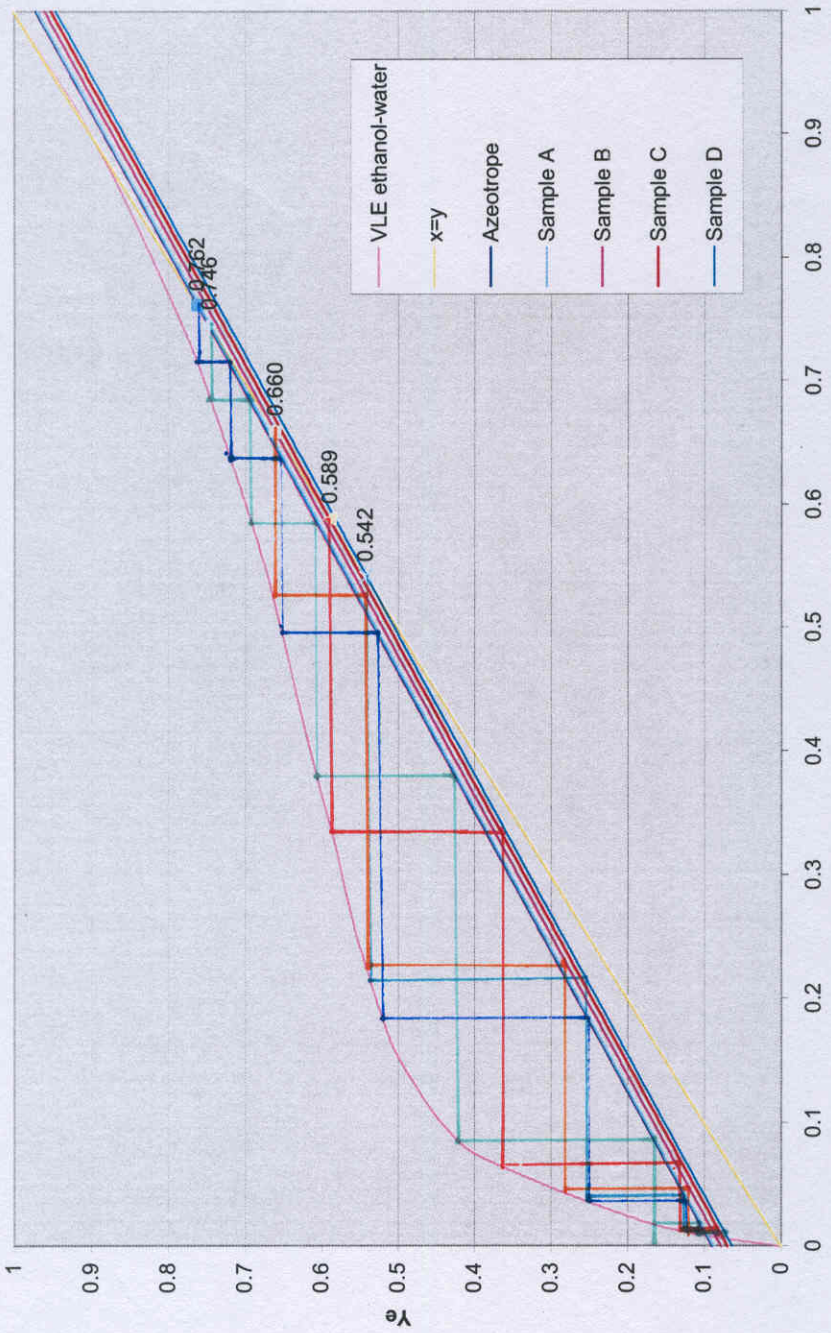


Fig A3.1 McCabe-Thiele diagram ethanol-water at 1 atm - Experiment 4, examination of operating lines generated under the assumption of CMO

APPENDIX A4 Raw Data: Experiment 1

Date 5/25/2007

Purpose: Batch distillation of Ethanol/water mixture with insulation around column.

x_{BStart} 6 wt% ethanol:94 wt% water

L/D 0.2

Energy Balance

Time hh:mm	Time Interval hh:mm	P_{steam} psig	$T_{steamin}$ °C	$m_{condensate}$ g	$t_{condensate\ collected}$ min	Steam Flowrate g/min	T_{cwin} °C	T_{cwout} °C	Cooling water Flowrate %	Cooling water Flowrate gal/min
9:58	0	8.0								
10:51	0:53	8.0	110.3	68.1	0.50	136.20	65.00	72.00	90	1.80
11:06	0:15	8.0	110.3				65.00	72.00	90	1.80
11:56	0:50	8.0	110.3				65.00	72.00	90	1.80
12:36	0:40	8.0	110.3				65.00	72.00	90	1.80
12:50	0:54									
13:09	0:33									
13:21	0:31									
13:30	0:21									

$t_{azeotrope}$ 1:45

$t_{startup}$ 0:53

$t_{operating}$ 3:49

t_{exp} 4:42

		Bottoms		Distillate					
Time hh:mm	Time Interval hh:mm	T_B °C	x_B mol%	T_{Top} °C	m_D g	$t_{distillate\ collected}$ min	D g/min	x_D mol%	Comments
9:58	0								
10:51	0:53	93.5		77.3			25.44		Start Distillate Flow (R = 7.5)
11:06	0:15	95.6		77.3			31.43		Distillate collected for entire time interval
11:56	0:50						22.80		Distillate collected for entire time interval
12:36	0:40								TD increasing; leaving azeotrope
12:50	0:54	97.9		78.1			14.06		
13:09	0:33	98.1		79.0					
13:21	0:31			80.0			14.06		
13:30	0:21			81.0					Stop Distillate Flow and Steam

APPENDIX A5 Raw Data: Experiment 2

Date 5/30/2007

Purpose: Batch distillation of Ethanol/water mixture with insulation around column.

x_{BStart} 3 wt% ethanol:97 wt% water

L/D 7.5

		Energy Balance								
Time hh:mm	Time Interval hh:mm	P_{steam} psig	$T_{steamin}$ °C	$m_{condensate}$ g	$t_{condensate\ collected}$ min	Steam Flowrate g/min	T_{cwin} °C	T_{cwout} °C	Cooling water Flowrate %	Cooling water Flowrate gal/min
11:10	0	10	112.9	64.44	0.41	156.98	67.00	75.00	90	1.80
11:58	0:48	10	113.1	59.41	0.45	133.26	67.00	74.00	90	1.80
12:10	0:12	10	113.0	62.75	0.41	151.33	67.00	74.00	90	1.80
12:22	0:12	10	113.1				66.50	75.00	90	1.80
13:41	1:19	10	112.9				67	75.00	90	1.80
14:27	0:46	10	113.1	45.60	0.33	136.80	66.50	75.00	90	1.80
15:22	0:55	10	113.1	43.95	0.43	102.93	66.50	75.00	90	1.80
15:59	0:37									
16:20	**0:21**									
16:42	**0:22**									
16:55	**0:13**									

$t_{azeotrope}$ 3:49

$t_{startup}$ 1:00

$t_{operating}$ 4:45

t_{exp} 5:45

		Bottoms		Distillate					
Time hh:mm	Time Interval hh:mm	T_B °C	x_B mol%	T_{Top} °C	m_D g	$t_{distillate\ collected}$ min	D g/min	x_D mol%	Comments
11:10	0								
11:58	0:48	95.3		78.2					
12:10	0:12	96.5		77.7					
12:22	0:12	97.1		77.5	118.80	17.43	6.82		Start Distillate Flow (R = 7.5)
13:41	1:19	98.6		77.3	278	45.38	6.13		Distillate collected for entire time interval
14:27	0:46	98.9		77.3	162.83	30.03	5.42		Distillate collected for entire time interval
15:22	0:55	99.1		77.3	211.96	42.27	5.01		Distillate collected for entire time interval
15:59	0:37	99.1		77.5					
16:20	**0:21**	**99.1**		**77.8**	**16.54**	**3.23**	**5.12**		**Sample Taken**
16:42	**0:22**	**99.2**		**78.3**	**17.55**	**3.50**	**5.01**		**Sample Taken**
16:55	**0:13**	**99.2**		**78.8**	**16.02**	**3.50**	**4.58**		**Sample Taken then Stop Distillate Flow & Steam**

APPENDIX A6 Raw Data: Experiment 3

Date 5/31/2007

Purpose: Batch distillation of Ethanol/water mixture with insulation around column.

x_{BStart} 1% ethanol:99% water

L/D 7.5

Energy Balance

Time hh:mm	Time Interval hh:mm	P_{steam} psig	$T_{steamin}$ °C	$m_{condensate}$ g	$t_{condensate\ collected}$ min	Steam Flowrate g/min	T_{cwin} °C	T_{cwout} °C	Cooling water Flowrate %	Cooling water Flowrate gal/min
9:15	0	10	111.9				67.00	75.00	90	1.80
10:40	1:25	10	113.3							
11:25	0:45	10	113.2							
11:34	**0:09**	**10**	**113.2**							
11:41	**0:07**	**10**	**113.2**							
11:50	**0:09**	**10**	**113.2**							
11:59	**0:09**	**10**	**113.2**							
12:11	**0:12**	**10**	**113.2**				67.00	75.00	90	1.80
12:15	0:04									
12:25	0:10									
13:25	1:00									
13:44	0:19									

$t_{azeotrope}$ **0:45**

$t_{startup}$ **1:25**

$t_{operating}$ **1:31**

t_{exp} **2:56**

$t_{recharge}$ **1:29**

		Bottoms		Distillate					
Time hh:mm	Time Interval hh:mm	T_B °C	x_B mol%	T_{Top} °C	m_D g	$t_{distillate\ collected}$ min	D g/min	x_D[1] mol%	Comments
9:15	0								Steam On
10:40	1:25	98.6		77.9					Start Distillate Flow (R = 7.5)
11:25	0:45	98.9		78.3	267.83	43.44	6.17		Distillate collected for entire time interval
11:34	**0:09**	**99.0**		**79.0**	**22.00**	**3.00**	**7.33**	**0.712**	
11:41	**0:07**	**99.1**		**79.7**	**15**	**2.05**	**7.32**	**0.660**	
11:50	**0:09**	**99.2**		**80.5**	**14.25**	**2.02**	**7.07**	**0.621**	
11:59	**0:09**	**99.2**		**82**	**13.54**	**2.03**	**6.66**	**0.567**	
12:11	**0:12**	**99.2**		**85**	**12.75**	**2.15**	**5.93**	**0.480**	**Stop Distillate Flow**
12:15	0:04								Stop Steam
12:25	0:10								Empty Still
13:25	1:00	62.6							Charge Start - Water only
13:44	0:19	26.1							Charge Complete - Water only

Total Distillate Collected	x_{DTotal}	**x_{DTotal}**	Total Ethanol Collected	Starting Ethanol In Bottoms	**Recovery of Ethanol[2]**			Total Distillate Collected	D_{avg}	t_{oper}
g	wt%	**mol%**	g	g	**%**			g	g/min	min
345.37	88.8%	**75.6%**	306.69	525.3	**58%**			345.37	6.75	51

[1] molar fractions determined for Temperature-composition diagram

[2] %recovery is not corrected because the final collection of distillate was spilled

APPENDIX A7 Raw Data: Experiment 4

Date 6/8/2007

Purpose: Batch distillation of Ethanol/water mixture without insulation around column.

x_{BStart} 1% ethanol:99% water

L/D 7.5

Energy Balance

Time hh:mm	Time Interval hh:mm	P_{steam} psig	$T_{steamin}$ °C	$m_{condensate}$ g	$t_{condensate\ collected}$ min	Condesate Flowrate g/min	T_{cwin} °C	T_{cwout} °C	Cooling water Flowrate %	Cooling water Flowrate gal/min
11:50	0	10	112.8							
12:15	0:25	10	112.8	59.42	0.51	117.62	67.00	75.00	90	1.80
12:39	0:24	10	112.8							
12:54	0:15	10	112.8							
13:05	0:11	10	112.8	60.51	0.51	119.66	67.00	75.00	90	1.80
13:10	0:05	10	112.8							
13:15	0:05									

Time hh:mm	Time Interval hh:mm	Q_c kJ/min	H_g J/g	H_f J/g	Q_r kJ/min	h_D J/g	Dh_D kJ/min	Q_{loss} kJ/min
11:50	0							
12:15	0:25	-228.34	2705.50	419.00	268.95	230.12	-1.45	-42.06
12:39	0:24							
12:54	0:15							
13:05	0:11	-233.63	2705.50	419.00	273.61	230.12	-1.15	-41.13
13:10	0:05							
13:15	0:05							

$t_{azeotrope}$ 0:25

$t_{startup}$ N/A

$t_{operating}$ 1:25

t_{exp} 1:25

Time hh:mm	Time Interval hh:mm	Bottoms T_B °C	Bottoms x_W mol%	Distillate T_{Top} °C	Distillate $T_{top,\ corrected}$ °C	Distillate m_D	Distillate $t_{distillate\ collected}$ min	Distillate D g/min	Distillate x_D mol%	Distillate x_D wt%	Comment
11:50	0	98.9	0.0042	77.6	78.4	20.45			0.762		Start Distillate Fl
12:15	0:25	98.9	0.0042	77.9	78.7	15.81	2.51	6.30	0.746	0.880	7.5)
12:39	0:24	99.1	0.0034	78.9	79.7	14.58	2.51	5.80	0.660		
12:54	0:15	99.1	0.0034	80.5	81.3	13.50	2.51	5.38	0.589		
13:05	0:11	99.1	0.0034	82	82.8	12.57	2.51	5.01	0.542	0.751	
13:10	0:05	99.1		83.2							Stop Distillate Fl
13:15	0:05										Stop Steam

Total Distillate Collected	x_{DTotal}	**x_{DTotal}**	Total Ethanol Collected	Starting Ethanol In Bottoms	**Recovery of Ethanol**
g	wt%	mol%	g	g	**%**
465.56	80.6%	**61.9%**	375.2	525.3	**71.4%**

x_D	x_W	$-\frac{1}{x_D - x_w}$	Area
0.76	0.0087	1.327	
0.60	0.0075	1.688	
0.50	0.0062	2.025	
0.40	0.0051	2.532	
0.30	**0.0039**	3.377	
0.20	0.003	5.076	
0.10	0.0015	10.152	
0.082	0.0012	12.376	-0.0194
0.055	**0.0008**	18.450	-0.0265
0.05	0.0007	20.284	

F	W_{final}	D_{final}	$x_{D,\ final}$	$x_{D,\ avg}$	$x_{W,\ final}$	% recovery
mol	mol	mol				
2.9	2.84	0.06	0.082	0.1317	0.0012	70%
2.9	2.82	0.08	0.055	0.11	0.0008	80%

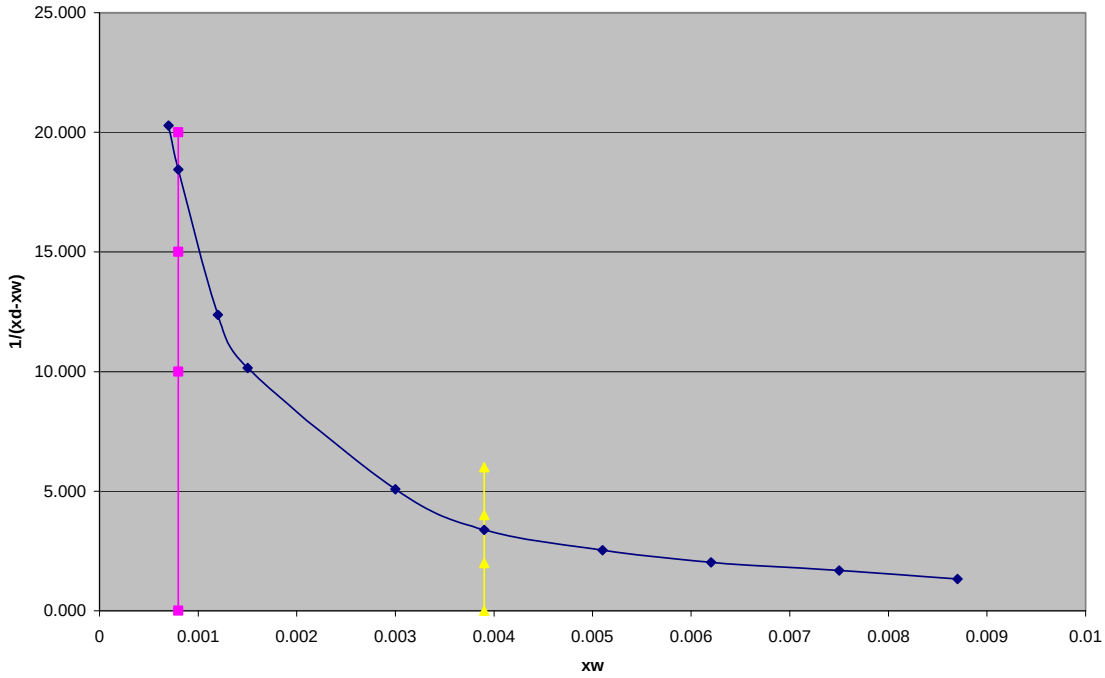


Fig. A7.1 Rayliegh equation analysis

APPENDIX A8 Raw Data: Experiment 5

Date 6/6/2007

Purpose: Verify stability of x_D versus time at Total Reflux outside of the azeotrope - distillation of Ethanol/water mixture with insulation around column.

x_{BStart} 1% ethanol:99% water

L/D Total Reflux

Time hh:mm:ss	Time Interval hh:mm:ss	T_{Top} °C	Comments
12:12:00	0	77.6	Start Distillate Flow (R = 7.5)
12:27:00	0:15:00	77.7	
12:31:00	0:04:00	77.8	
12:33:00	0:02:00	77.9	
12:37:00	0:04:00	78.0	
12:39:00	0:02:00	78.1	
12:41:00	0:02:00	78.2	
12:43:00	0:02:00	78.3	
12:45:00	0:02:00	78.4	
12:46:00	0:01:00	78.5	
12:47:00	0:01:00	78.6	Stop Distillate Flow
12:50:00	0:03:00	78.7	
13:01:00	0:11:00	78.8	
13:17:00	0:16:00	78.8	
13:30:00	0:13:00	78.9	
13:45:00	0:15:00	78.9	
14:00:00	0:15:00	79.0	Start Distillate Flow (R = 7.5)
14:02:00	0:02:00	79.1	
14:03:00	0:01:00	79.2	
14:04:00	0:01:00	79.3	
14:05:00	0:01:00	79.4	
14:05:40	0:00:40	79.6	
14:07:00	0:01:20	79.7	
14:07:30	0:00:30	79.8	
14:10:00	0:02:30	79.9	
14:10:20	0:00:20	80.0	
14:10:40	0:00:20	80.1	
14:11:20	0:00:40	80.2	
14:11:45	0:00:25	80.3	
14:12:25	0:00:40	80.4	
14:12:45	0:00:20	80.5	
14:13:00	0:00:15	80.6	
14:13:50	0:00:50	80.7	
14:14:50	0:01:00	80.8	
14:15:05	0:00:15	80.9	
14:15:30	0:00:25	81.0	
14:15:45	0:00:15	81.1	
14:16:10	0:00:25	81.2	
14:16:25	0:00:15	81.3	
14:16:40	0:00:15	81.4	
14:17:30	0:00:50	81.5	
14:17:40	0:00:10	81.6	
14:17:55	0:00:15	81.7	
14:18:15	0:00:20	81.8	
14:18:40	0:00:25	81.9	

14:19:05	0:00:25	82.0	
14:19:15	0:00:10	82.1	
14:19:45	0:00:30	82.2	
14:20:10	0:00:25	82.3	
14:20:55	0:00:45	82.4	
14:21:05	0:00:10	82.5	
14:21:30	0:00:25	82.6	
14:21:45	0:00:15	82.7	
14:22:05	0:00:20	82.8	
14:22:20	0:00:15	82.9	
14:22:35	0:00:15	83.0	
14:22:55	0:00:20	83.1	
14:23:15	0:00:20	83.2	
14:23:30	0:00:15	83.3	
14:24:10	0:00:40	83.4	
14:24:30	0:00:20	83.5	
14:24:50	0:00:20	83.6	
14:25:00	0:00:10	83.7	
14:25:25	0:00:25	83.8	
14:25:30	0:00:05	83.9	
14:25:40	0:00:10	84.0	
14:25:55	0:00:15	84.1	
14:26:30	0:00:35	84.2	
14:26:45	0:00:15	84.3	
14:27:00	0:00:15	84.4	
14:27:20	0:00:20	84.5	
14:27:30	0:00:10	84.6	
14:27:35	0:00:05	84.7	
14:28:00	0:00:25	84.8	
14:28:35	0:00:35	84.9	
14:28:40	0:00:05	85.0	
14:28:45	0:00:05	85.1	

Time hh:mm	Time Interval hh:mm	T_B °C	Comments
14:24	0	98.5	Start Drain Bottoms
14:50	0:26	95.3	Still Empty
15:50	1:00	60.2	Start Charging - Water Only
16:05	0:15	23.6	Charging Complete - Water Only

Total Downtime Time **1:41**

Total Distillate Collected g	x_{DTotal} wt%	Total Ethanol Collected g	Starting Ethanol In Bottoms g	**Recovery of Ethanol** **%**
550.72	87.6%	482.43	525.3	**91.8%**

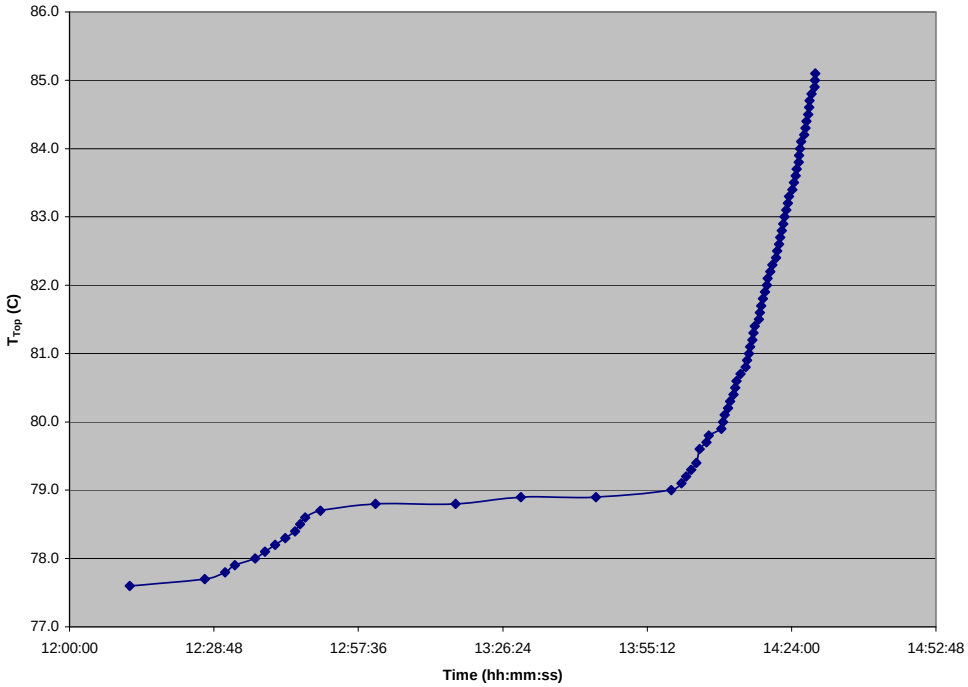


Fig. A8.1 Experiment 5 - Temperature versus time

APPENDIX A9 VLE Data ethanol-water at 1 atm

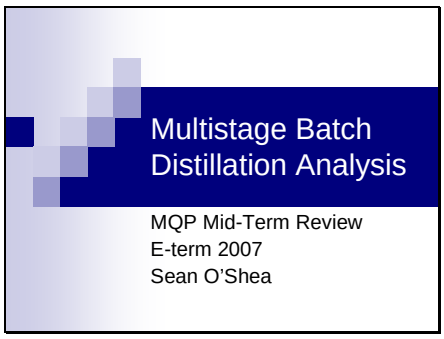
VLE Data Ethanol-Water @ 1 atm

The molar data is from Perry 13-12.

Temperature	Mole Fraction		Mass Fraction (g/g)		Volume Fraction (mL/mL)	
(C)	Liquid (x)	Vapour (y)	Liquid (x)	Vapour (y)	Liquid (x)	Vapour (y)
100	0	0	0	0	0	0
95.5	0.019	0.17	0.0472	0.3437	0.0591	0.399
89	0.072	0.389	0.1657	0.6196	0.2012	0.6736
86.7	0.097	0.438	0.2147	0.6654	0.2573	0.716
85.3	0.124	0.47	0.2654	0.6943	0.3141	0.7422
84.1	0.166	0.509	0.3374	0.726	0.3923	0.7705
82.7	0.234	0.545	0.4381	0.7535	0.4971	0.7948
82.3	0.261	0.558	0.4743	0.7635	0.5334	0.8036
81.5	0.327	0.583	0.5544	0.7811	0.6119	0.8189
80.7	0.397	0.612	0.6269	0.8014	0.6804	0.8365
79.8	0.508	0.656	0.7252	0.8301	0.7698	0.8609
79.7	0.52	0.66	0.7346	0.8322	0.7782	0.8628
79.3	0.573	0.684	0.7745	0.847	0.8132	0.8753
78.74	0.676	0.739	0.8423	0.8784	0.8713	0.9015
78.24	0.747	0.782	0.8831	0.9014	0.9055	0.9206
78.15	0.894	0.894	0.9558	0.9558	0.9648	0.9648
78.3	1	1				

APPENDIX A10 MQP Mid-Term Review Presentation

Slide 1



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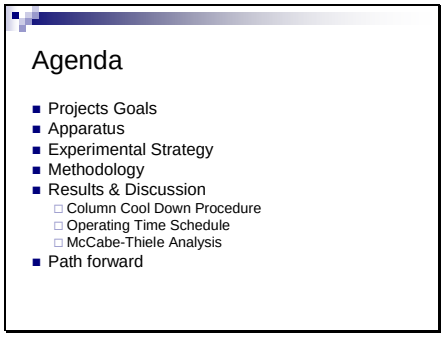

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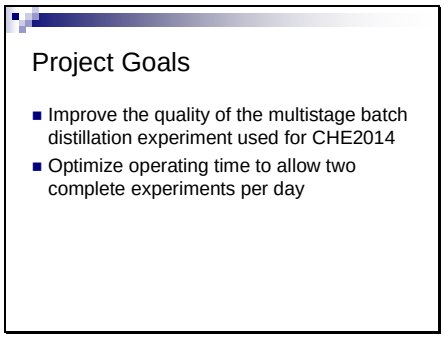
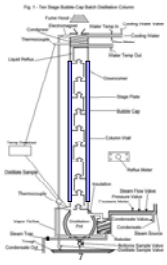


Slide 4

Apparatus



Slide 5

Experimental Strategy

Process Change	Result
Maximize P_{steam}	Minimize $t_{startup}$
Reduce $x_{B_{intial}}$	Minimize t_{oper} at Azeotrope
Determine Balanced R	Reasonable Sampling Time at Constant D
Minimize Cool-Down Time of Column	Run Minimum of 2 Experiments/Day

Slide 6

Experimental Strategy

Process Variable	Previous MQP	Current MQP
$x_{B_{intial}}$	0 – 9%	0 – 3%
P_{steam}	5 – 8%	10 – 14* psig
R	0.2 – 0.4	$1.5R_{min} = 7.5$

Slide 7

Experimental Strategy

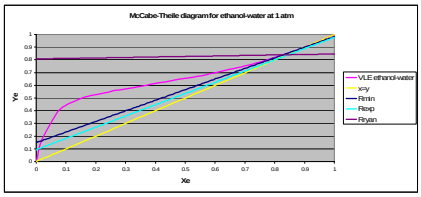
Determination of R_{min}

"R_{min} is defined as the external reflux ratio at which the desire separation could just be obtained with an infinite number of stages."
– Philip C. Wankat

"R_{min} is determined from pitch points between the operating line and VLE data."
– Philip C. Wankat

Slide 8

Experimental Strategy



Slide 9

Methodology

Process Variable	Experiment 1	Experiment 2	Experiment 3
$x_{B_{intial}}$	~6%	3%	1%
P_{steam}	8 psig	10 psig	10 psig
R	0.2	7.5	7.5

Slide 10

Results and Discussion

Process Variable	Experiment 1	Experiment 2	Experiment 3
$x_{B_{initial}}$	~6%	3%	1%
P_{steam}	8 psig	10 psig	10 psig
R	0.2	7.5	7.5
$t_{startup}$	53 min	48 min	~60 – 90 min*
t @ azeotrope	3 hrs 51 min	3 hrs 59 min	**45 min**
# of samples after azeotrope	0	3	5
t_{oper}	6 hrs	6 hrs	**3 hrs**
% recovery	N/A	N/A	**87%**

Slide 11

Column Cool Down Procedure

- Drain bottoms into sewer
- Air cool for 1 hr (T_B ~ 60°C)
- Recharge still with required water only
- Add ethanol when T_B < 30°C

Slide 12

Operating Time Schedule

Steam On – Column startup	8:00 AM
Start R – Begin Experiment	9:00 AM
Steam Off, Empty Still, & Air Cool	10:30 AM
Charge Still	11:40 AM
Steam On – Column startup	12:00 PM
Start R – Begin Experiment	1:00 PM
Steam Off, Empty Still, & Air Cool	2:30 PM
Charge Still	3:40 PM

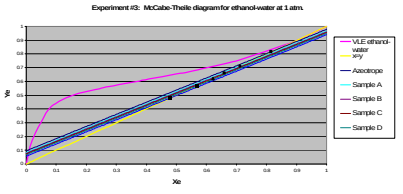
Slide 13

McCabe-Thiele Analysis

- Data Assumptions
  - Low volume Hydrometer is out for repair
  - All x_D determined from "Enthalpy-concentration diagram for ethanol-water at a pressure of 1 atm". – Phillip C. Wankat
  - Azeotrope concentration measured from distillate collected until T_T changes

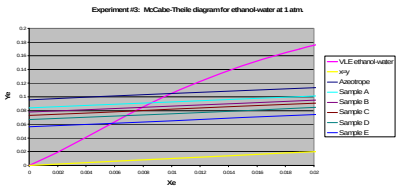
Slide 14

McCabe-Thiele Analysis



Slide 15

McCabe-Thiele Analysis



Problem: it is not possible to achieve $x_{B_{measured}}$

Hypothesis: CMO is not valid due to heat loss at the reboiler & top stage.

Slide 16

Path forward

- Connect permanent feed line of DI water to still pot
- Rerun experiment #3 to make recommendations on how to effectively run the experiment vs. number of students required
- Update experiment documentation to reflect new procedure and parameters
- Complete written MQP report

REFERENCES

[1] Philip C. Wankat *Separation in Chemical Engineering: Equilibrium-Staged Separations,* Elsevier Publishing Co., Inc., pp. 117

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[3] Philip C. Wankat *Separation in Chemical Engineering: Equilibrium-Staged Separations,* Elsevier Publishing Co., Inc.,., pp. 118

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[9] R. E. Treybal, *Mass-Transfer Operations*, 2nd Editon, McGraw Hill, 1968, pp. 337-39